



## Evaluation of imazethapyr leaching in soil under natural rainfall conditions

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### ABSTRACT

Though herbicides are designed as biologically active but often their residues are found in crop produce and water. Herbicides may reach to ground water through runoff, drift, untargeted spray and heavy rain. Therefore, leaching behaviour of imazethapyr, was evaluated in soil columns under natural rainfall conditions. Imazethapyr was applied at 100 and 200 g/ha on the soil columns. Columns were arranged randomly and allowed to receive approximately 850 mm rain for three months. Soil and leachates were analyzed for imazethapyr residues. Maximum concentration of imazethapyr was recovered from 0-10 cm depth. Results indicated that imazethapyr could leach in clay loam soil up to the depth of 70 cm.

**Key words:** Clay soil, Column, Imazethapyr, Leaching, Movement, Rainfall

Use of herbicides for weed control has increased with the introduction of modern intensive crop management practices. Increasing use of herbicides may pose serious environmental problems through off site movement and leaching, which must be controlled to minimize harmful effects on non-targeted organism. Ultimately, leaching and transport of herbicides may not only result in low efficacy, but also in groundwater contamination (Ritter *et al.* 1996, Sondhia 2008a).

Imazethapyr belongs to imidazolines group having a toxicity class of III and used as a selective herbicide. It is applied as pre-plant incorporated, pre-emergence, and post-emergence to control grasses and broad-leaf weeds. Imazethapyr persist longer in acid than in alkaline soils. Organic matter and pH significantly affect the imazethapyr behaviour in soil. However, imazethapyr does not readily leach under field condition but few authors reported leaching of imazethapyr below 25 cm in four months in acidic soil under laboratory studies (Basham *et al.* 1987). Residues of imazethapyr are detected in stream and river water in Midwestern US at concentrations above the maximum residue limits in 71% of samples (Battaglin *et al.* 2000).

Imidazolinone herbicides are generally weakly adsorbed to soil (Basham *et al.* 1987, McDowell *et al.* 1997, Sondhia 2008b). Few studies have shown that imidazolinone adsorption is not affected by clay or organic matter content (Bresnahan *et al.* 2000). However, few other studies have indicated that adsorption increases with increasing clay and organic matter content (Loux *et al.* 1989, Battaglin *et al.* 2000).

In India, despite of the extensive use of herbicides, information on leaching potential of herbicides under high rainfall conditions is meager. Therefore, the present study was undertaken with the objective to study the mobility and leaching potential of imazethapyr under natural rainfall conditions.

### MATERIALS AND METHODS

Leaching experiment was conducted at Residue Laboratory of Directorate of Weed Science Research, Jabalpur at ambient temperature (27 to 32°C) as described by Sondhia and Yaduraju (2005) in polyvinyl chloride (PVC) columns (10 cm internal diameter and 90 cm long).

Herbicide free surface soil samples (0-20 cm) from the surrounding area of the Directorate of Weed Science Research were collected, air-dried and passed through a 3 mm sieve. Soil characteristic was clay, 31.27; silt, 11.15; sand, 56.39% with 7.2 pH and 0.82% organic carbon. Approximately 9 kg soil was placed in each PVC column. The bottoms of the columns were closed with a double layer of cheese cloth. Columns were sequentially filled with soil from the bottom with 3 cm of sand and 86 cm of dry soil. The surface of each column was then covered with sand (3 cm) and filter paper disks were placed on top of the each column to assist uniform dispersion of the water across the column surface. A perforated PVC cap connected to a funnel with polyethylene tubing was attached to the bottom of the each column to collect the leachates into 500 ml flasks. Commercial grade of imazethapyr (10% SL) were used in leaching experiment.

Imazethapyr was dissolved in distilled water and simultaneously applied to the surface of the column with

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a pipette at dose of 100 and 200 g/ha. Physico-chemical property of imazethapyr is given in Table 1. The columns were arranged randomly under field condition to receive the water through natural rainfall. Treatments were replicated three times. A set of soil columns receiving respective amount of rain only served as control. Columns were arranged in a completely randomized design and each treatment combination was replicated three times. Water eluting from the column were collected in flask and stored at 5°C for herbicide analysis. After 13 weeks, soil columns were cut into two equal halves and the soil was sampled in 10 cm segments. The segments from same column were pooled for use in analyzing residues.

**Table 1. Physico-chemical properties of imazethapyr**

Molecular weigh	289.33
Molecular formula	C <sub>15</sub> H <sub>19</sub> N <sub>3</sub> O <sub>3</sub>
Formulation	SI 10%
Solubility	0.014 g/ml at mg/L (25 °C)
Vapour pressure	1.33 X 10 <sup>-02</sup> mPa at 25 °C
Henry constant	1.30 X 10 <sup>-02</sup> Pa m <sup>3</sup> /mol
Partition coefficient LogP <sub>ow</sub>	1.49

Imazethapyr residues were determined as described by Sondhia (2008b). Twenty five g representative soil samples were taken in 250 ml Erlenmeyer flask, and extracted with 50 ml of 0.5 N NaOH for 1 hour in a horizontal shaker (repeated twice). Flask were washed with 50 mL of methanol, shaken vigorously and adjusted the pH to 2 with 6 N hydrochloric acid. The content was transferred to a 250 ml separatory funnel and partitioned with methylene chloride (50 ml twice). The lower methylene chloride layer was collected and combined. The organic layer was dried on anhydrous Na<sub>2</sub>SO<sub>4</sub> and passed through activated charcoal. The solvent evaporated to dryness on rotary evaporator at 40°C. Finally residues were dissolved in 5 mL of methanol.

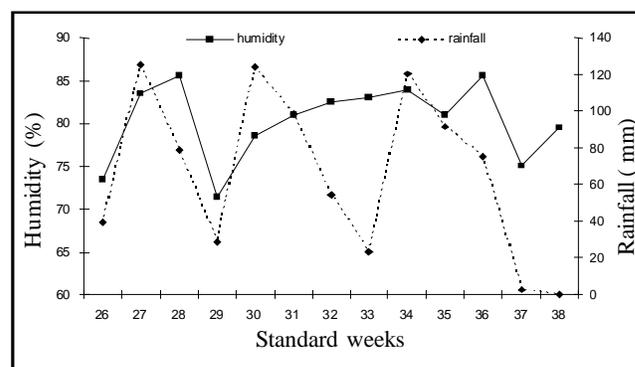
25 ml water samples were passed through filter paper No 2 and pH was adjusted to 2 with 6 N hydrochloric acid and separated with methylene chloride (50 ml twice) in a 250 ml separatory funnel. The lower methylene chloride layer was collected and combined, processed and concentrated as discussed above for the soil samples.

Imazethapyr reference analytical standard was obtained from Ehrenstorfer GmbH, Germany. All the other chemicals and solvents used in the study were of analytical grade obtained from Merck, Germany. Imazethapyr content in soil and leachates at various depths were determined by Shimadzu HPLC, coupled to diode array detec-

tor using Phenomenex C-18 (ODS) column (250 x 4.6 mm) and methanol: water (70:30 v/v) as mobile phase at a flow rate of 0.8 ml/min. Detection of imazethapyr in soil and leachates was carried out at 250 nm. 20 µl of the aliquot of samples and standard was injected into the column through fixed loop Rheodyne injector using micro syringe. The retention time of imazethapyr was found approximately 3.45 minutes. At the limit of detection 0.001µg/ml the signal to noise ratio was 3:1. Imazethapyr content in the soil and leachates was determined by comparing peak area of samples with standards.

## RESULTS AND DISCUSSION

Columns placed in field received approximately 850 mm natural rain during the experimental period, viz. Jun 2007 to September 2008 (Fig. 1).



**Fig. 1. Variation in humidity and rainfall during experimental period from July to September 2007**

Significant difference of imazethapyr content was observed at 0-10, 10-20 and 20-30 cm depth at P=0.05 level (Table 2) in both the application rates. However, at 30-40, 40-50, 50-60, 60-70, and 70-80 cm soil depths difference of imazethapyr content were non significant. Results showed that 0.119 µg/g of imazethapyr was found in 0-10 cm depth, however 0.039, 0.0210, 0.160, 0.013, 0.011, and 0.010 µg/g imazethapyr content was detected in 10-20, 20-30, 30-40 40-50, 50-60 and 60-70 cm soil depth in 100 g/ha treated soil columns (Table 2).

Approximately 48% imazethapyr was found distributed in 0-10 cm depth and 4.49 and 4.08% imazethapyr could leach to the 50-60 and 60-70 cm soil depths which showed high adsorption of imazethapyr at the surface soil (0-10 cm) as compared to sub-surface soil (50-70 cm). However, 15.92, 8.57, 6.53 and 4.49% of imazethapyr was recovered from 20-30, 30-40, 40-50 and 50-60 cm depths in 100 g/ha treated soil columns (Fig. 2).

**Table 2. Herbicides content at different depths in soil column that received 850 mm rainfall**

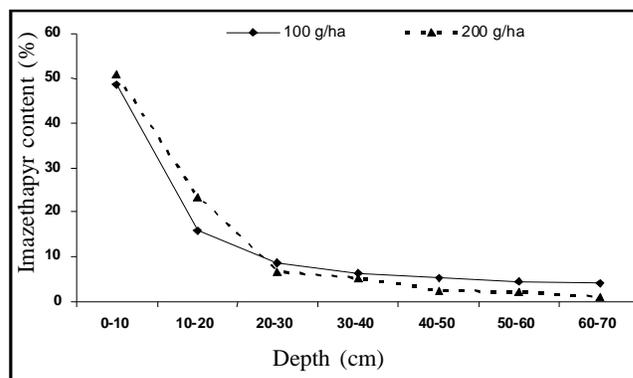
Soil depth (cm)	Imazethapyr content ( $\mu\text{g/g}$ )*	
	100 g/ha	200 g/ha
0-10	0.119 a** (+0.03***)	1.411 a (+0.01)
10-20	0.039 b ( $\pm 0.01$ )	0.654 b ( $\pm 0.02$ )
20-30	0.021 a ( $\pm 0.01$ )	0.182 a ( $\pm 0.01$ )
30-40	0.016 a ( $\pm 0.03$ )	0.151 a ( $\pm 0.04$ )
40-50	0.013 a ( $\pm 0.01$ )	0.059 a ( $\pm 0.02$ )
50-60	0.011 a (+0.01)	0.067 a (+0.01)
60-70	0.010 a ( $\pm 0.01$ )	0.032 a ( $\pm 0.01$ )
70-80	<0.010	<0.010

\* Average of four replications, \*\* Within each column and for each depth mean followed by similar letter are not significant according to LSD ( $P=0.05$ ), \*\*\* Standard deviation

Whereas, approximately 50% imazethapyr was found distributed in 0-10 cm depth in soil column where imazethapyr was applied at 200 g/ha rate, however 23.58% imazethapyr could leach to the 10-20 cm soil depths. At 20-30, 30-40, 40-50, 50-60 and 60-70 cm depths, 6.56, 5.44, 2.41, 2.13 and 1.15% of imazethapyr was recovered from 200 g/ha treated soil columns (Fig. 2) that showed high adsorption of imazethapyr at surface soil (0-10 cm) as compared to subsurface soil (20-70 cm) at higher application rate. These findings were in conformity of Battaglin *et al.* (2000) and indicated that imazethapyr may leach in clay soil up to the depth of 80 cm if soil received rainfall of 850 mm. Organic matter and pH significantly affect the imazethapyr behaviour in soil (Mangles 1991). Some authors reported leaching of imazethapyr below 25 cm in four months in acid soil in lab studies and detected imazethapyr residues in stream and river water in Midwestern US at concentrations above the maximum residue limits in 71% of samples (Basham *et al.* 1986, Battaglin *et al.* 2000).

Results obtained by this study revealed that imazethapyr was mobile in soil columns and can leach by irrigation water. The highest concentration of imazethapyr was found at 0-10 cm depth in both the application rates and it was higher in 100 g/ha application rate than 200 g/ha rate. The presence of organic matter constitutes an impediment for imazethapyr movement because of its high adsorption capacity. Thus maximum amount of applied imazethapyr was retained from 0-10 cm depth followed by 0-20 cm depth in both application rates (Fig. 2).

Recovery of imazethapyr from the leachates was low and only 1.75 and 1.13% of imazethapyr was recovered

**Fig. 2. Distribution of imazethapyr at different depths in soil column**

from leachates where imazethapyr was applied at 100 and 200 g/ha doses, respectively. Soil moisture, temperature, organic matter, pH, texture and rains are the main factors that determine mobility of herbicide (Vanwyk and Reinhardt 2001, Sondhia 2008). The data generated here indicated mobility of imazethapyr up to 70 cm soil depth under continuous rainfall, which is significant in terms of ground water contamination. Leaching of some other classes of herbicides through soil is also reported by other worker. James *et al.* (1999) also reported detectable and significant amounts of metsulfuron-methyl into the 50–100 mm soil layer. Sondhia (2008b) reported that metsulfuron is more mobile in a sandy soil than in clay soil and found that metsulfuron mobility in soil was increased with increasing application rate.

Combination of chemical, biological, physical and environmental factors may operate at different level in influencing the degradation of herbicides. Some researchers recommend re-cropping periods of up to 6-34 months for imazethapyr due to leaching and persistence and reported that imazethapyr has a rapid initial phase of degradation, followed by a slower second phase leading to long term persistence especially in clay soil Bresnahan *et al.* (2000).

Imidazolinone herbicides are generally weakly adsorbed to soil. Few studies have shown that imidazolinone adsorption is not affected by clay or organic matter content (Goetz *et al.* 1986, Mangles 1991, Gan *et al.* 1994, McDowell *et al.* 1997). However, few other studies have indicated that adsorption increases with increasing clay and organic matter content (Stougaard *et al.* 1990). Solubility of imazethapyr is high in water (140 mg/ml), hence able to leach from the top-soil to deeper in the soil profile and thus may not be available in the surface soil (0-20 cm). In general, depth wise mean

concentration of all herbicides followed the decreasing pattern. The top soil acts as the inoculum for the system and is likely to vary in terms of its physical, chemical, and microbiological characteristics from one form to another. The presence of organic matter constitutes an impediment for herbicides movement because of its high adsorption capacity.

Downward herbicide movement is influenced by characteristics of the soil. In addition, certain soil microorganisms and living weeds can sometimes metabolize absorbed herbicides, rapidly or gradually altering those to non-phytotoxic forms that may have different leaching characteristics. Downward movement is most likely with chemicals that do not degrade quickly and do not adsorb strongly to clay or organic matter. The potential for ground-water contamination is greatest with such chemicals when heavy rain comes soon after application, or where spills occur. Increasing the time interval between herbicide application and the incidence of rainfall reduced the amounts of herbicides found in the leachates. Pesticide leaching from zone of the soil application is also dependent on amount of rainfall or irrigation received. Low recovery of imazethapyr from soil and water may be due to formation of bound residues, secondary metabolites and dissipation of imazethapyr from soil.

Although, soil moisture generally has an effect on the adsorption of herbicides to soil, it is unlikely to dominate herbicide transport unless this transport is very rapid. Thus, in leaching study in soil column, leaching could have been slow initially and gradually increased as water penetrated deeper. The data generated indicated that imazethapyr can move up to 70 cm in soil profile under continuous and high rainfall conditions.

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